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     (FILE 'HOME' ENTERED AT 17:22:12 ON 18 DEC 2001)

     FILE 'CA, BIOSIS, MEDLINE' ENTERED AT 17:23:47 ON 18 DEC 2001
L1          713 S HUMULONE?
L2          363 S LUPULONE?
L3          246 S L1 (P) L2
L4      1105627 S ANTIOXIDANT? OR VITAMIN? OR MINERAL?
L5          246 S L3 AND L3
L6            2 S L3 AND L4
L7       352045 S PHARMACEUTICAL?
L8            2 S L7 AND L3
L9         4822 S HOPS?
L10         122 S L4 AND L9
L11         103 S L4 (P) L9
L12           3 S L7 AND L11

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L9: Entry 29 of 35 File: USPT Jan 9, 1979

DOCUMENT-IDENTIFIER: US 4133903 A
TITLE: Process for preparing bitter beverages

Brief Summary Text (3):
Cynaropicrin is a known plant ingredient with intense bitter taste. Plants and vegetable matter containing bitter compounds as well as bitter principles isolated therefrom have been used for considerable time as antipyretics. Extracts containing bitter principles as well as isolated bitter substances are used today mainly for the preparation of bitter beverages stimulating appetite and digestion, primarily however to be consumed in the luxury food and drinks trade. Besides beer, containing bitter hop extract, there have appeared bitter beverages with higher alcohol contents such as aperitifs, liqueurs and others, containing in addition to flavouring and sweetening agents extracts from different species of the plant family Compositae and the genus Citrus. Besides them, bitter tasting and more or less flavoured and sweetened carbonated waters (so called tonic waters have) became commercially very important. The main ingredients for bitter beverages of this kind are ingredients of the genus Citrus and especially quinine. Quinine has been used since the 17th century for combatting malaria fever. Quinine however exerts at higher doses relatively complex pharmacodynamic effects. Its use in beverages is therefore by no means harmless. For this reason the beverage industry has attempted to replace quinine by pharmacologically less harmful bitter compounds.

Detailed Description Text (14):
Such a base can further contain suitable dyes, additional essences, fragrances, bitter compounds etc. 5 kilo of the base are then mixed with 95 kg sugar solution (60%) to a syrup. This syrup is diluted in the bottle by a tenfold amount of carbonated water (water saturated by carbon dioxide).

L9: Entry 30 of 35 File: USPT Oct 31, 1978

DOCUMENT-IDENTIFIER: US 4123561 A
TITLE: Method for processing hops for brewing

Brief Summary Text (17):
(i) mixing hops or hop extracts containing alpha acids with one or more metallic oxides wherein the metal is divalent and suitable for use in food products, so as to intimately contact the oxide material with the alpha acids present in the hops; and

Detailed Description Text (5):
If desired, carbon dioxide can be fed in a continuous stream from pressurized cylinder 10 through regulating pump 12 into press 8 so as to form a blanket of the gas above the pellet die, and a flushing stream below the pellet die, during pellet formation. During the pelletizing operation, isomerization of the alpha acids in the hops powder begins to occur.

L9: Entry 28 of 35 File: USPT May 15, 1979

DOCUMENT-IDENTIFIER: US 4154865 A
TITLE: Method for processing hops for brewing

Abstract Text (1):
There is provided a method of processing hops or hop extracts for brewing in which hops and particularly the alpha acids in the hops are stabilized against deterioration and light sensitivity, the process broadly comprising isomerizing a substantial portion of the alpha acids in the hops and contacting said iso-alpha acids witha metallic hydride compound, the metal thereof being suitable for use in foods, until the reaction is substantially completed. In another aspect, the alpha acids present in the hops are converted to their reduced isomerized products which are desirable for brewing. The process is especially suitable for use in pelletizing operations.

Brief Summary Text (21):
(ii) contacting the hop extracts containing isomerized alpha acids with a metallic hydride compound suitable for use in foods under suitable conditions to stabilize said isomerized alpha acids.

Brief Summary Text (23):
Any of the methods for isomerizing alpha acids, such as described in the aforementioned patents, are suitable in accordance with the present invention. In a preferred method of isomerizing the alpha acid constituents of hops, it has been found advantageous to mix the hops or hop extracts with one or more metallic oxides wherein the metal is divalent and suitable for use in food products and wherein the mixing intimately contacts the oxide material with the alpha acids in the hops. Examples of suitable metallic oxides include calcium oxide, magnesium oxide or a mixture of calcium oxide and magnesium oxide.

Detailed Description Text (7):
If desired, carbon dioxide can be fed in a continuous stream from pressurized cylinder 10 through regulating pump 12 into press 8 so as to form a blanket of the gas above the pellet die, and a flushing stream below the pellet die, during pellet formation.

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L9: Entry 32 of 35 File: USPT Sep 23, 1975

DOCUMENT-IDENTIFIER: US 3908021 A
TITLE: Preparing a neutral tasting alcoholic base

Detailed Description Text (16):
After the second storage, the base is polish filtered and can be carbonated if desired to approximately 3.5 volumes of carbon dioxide.

CLAIMS:

6. A method of preparing a neutral tasting alcoholic base, comprising the steps of mixing a low soluble protein-low-kiln malt with water at a temperature in the range of 66.degree.C to 77.degree.C to provide a mash, said malt having a moisture content in the range of 5% to 6% by weight, a diastase value in the range of 150 to 240, and a soluble protein content based on total protein in the range of 30 to 37% by weight, maintaining the mash at said temperature for a period of time of 30 to 90 minutes to produce a wort, boiling the wort for a period of 10 to 40 minutes, mixing the boiled wort with an adjunct containing from 40% to 95% by weight of fermentable carbohydrate and an isomerized hop constituent to provide a fermentable extract, said hop constituent being present in an amount equivalent to at least 7 pounds of 4% alpha acid hops per 100 barrels of said fermentable extract, said adjunct being present on a solids basis in the amount of 70% to 95% by weight of the combined weight of said wort and said adjunct, adding to the extract prior to fermentation a water-soluble, food-compatible nitrogen-containing compound in an amount sufficient to provide a nitrogen content of 50 to 500 ppm in the extract, adding brewer's yeast to said extract, fermenting the extract to provide a fermented substrate, and thereafter removing the yeast from the substrate to provide a neutral-tasting alcoholic substrate.

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L9: Entry 35 of 35 File: DWPI

DERWENT-ACC-NO: 1972-47672T
DERWENT-WEEK: 197230

TITLE: Malt beverage - with champagne flavour brewed from sugar , malt, soya and acid composn

PATENT-ASSIGNEE:

ASSIGNEE	CODE
HAMM CO T	HAM N

PRIORITY-DATA: 1970US-0100521 (December 21, 1970)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
DE 2163263 A			000	
CA 944706 A	April 2, 1974		000	
US 3720517 A			000	

INT-CL (IPC): C12C 11/04

ABSTRACTED-PUB-NO: DE 2163263A
BASIC-ABSTRACT:

Naturally brewed malt beverage, with champgne-flavour, contains a CO2-satd. yeast-fermented alcoholic malt base, 0.379-7.57 1. of a 77 wt.% soln. of fermentable sugar, e.g. dextrose, cane- and/or invert sugar per 3.79 1. of base and 0.181-0.544 kg acid such as fumaric, pref. citric or mixt. per 117 1. of base. Aqs. fermentation medium contg. 2-20 wt.% fermentable carbohydrate, 1-25 wt.% malt, 0.1-0.3 wt.% hops or hop extract and 0.05-0.2 wt.% food quality material contg. >35 wt.% soluble protein or aminoacids is prepd.; boiled, fermented with brewer's yeast to form alcoholic aqs. base; admixed with the acid and sugar and satd. with CO2, esp. 2.5-4 vol/vol. base.

TITLE-TERMS: MALT BEVERAGE CHAMPAGNE FLAVOUR BREW SUGAR MALT SOY ACID

DERWENT-CLASS: D16

CPI-CODES: D05-B; D05-E;

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L9: Entry 27 of 35 File: USPT Jul 14, 1981

DOCUMENT-IDENTIFIER: US 4278012 A
TITLE: Plant for the extraction of hops by extraction with liquid carbon dioxide

Assignee Name (1):
The Distillers Company (Carbon Dioxide) Limited

Assignee Group (1):
The Distillers Company (Carbon Dioxide) Limited Reigate GB2 03

Abstract Text (1):
A method of preparing an extract from a material comprises contacting the material to be extracted with carbon dioxide in the liquid state to dissolve the matter to be extracted in the liquid carbon dioxide, heating the solution of extract to evaporate carbon dioxide from the solution of extract, compressing and condensing the evaporated carbon dioxide vapor to re-form liquid carbon dioxide, and re-cycling the liquid carbon dioxide to contact the material to be extracted. At least part of the heat evolved from the carbon dioxide vapor after its compression and during its change of state from vapor to liquid carbon dioxide is used to cause the change of state of the liquid carbon dioxide in the solution of the extract to evaporate the carbon dioxide from the solution of the extract.

Abstract Text (2):
A plant for the preparation of an extract of hops by extraction with liquid carbon dioxide is also disclosed together with modifications of the plant to enhance the recovery of hop oils or obtain a separate extraction of the hop oils.

Brief Summary Text (1):
This invention relates to a method of preparing, and a plant for the preparation of, a extract from a material by extracting the matter from the material using liquid carbon dioxide as the extracting agent and it has particular application to the extraction of the bittering principles from hops.

Brief Summary Text (2):
U.S. Pat. No. 3,477,856 discloses a general process for the extraction of flavours and flavour containing materials from a wide variety of different organic substances using liquid carbon dioxide as the extracting agent. The material to be extracted and the liquid carbon dioxide are passed through an extraction column in which the matter to be extracted is dissolved in the liquid carbon dioxide and the solution of the extracted matter in the liquid carbon dioxide is then passed to an evaporator in which the carbon dioxide is evaporated. The matter extracted remains in the evaporator and the carbon dioxide vapour is condensed in a further, independent, condenser and then re-cycled into contact with the matter to be extracted.

Brief Summary Text (3):
A considerable amount of work has been carried out to produce extracts of coffee and tea using carbon dioxide as the extracting agent but, in general, this has always involved the use of carbon dioxide as a super critical fluid. In particular, super critical carbon dioxide is used to prepare a de-caffeinated coffee. British Patent Specification No. 1,346,134 is a typical example of one of the various extraction processes. In this Specification, carbon dioxide which is super critical with respect to both pressure and temperature passes through an extraction column packed with ground coffee beans to extract the coffee oil containing the aroma constituents

of the coffee. After all of the coffee oil has been extracted, water is introduced into the system and the completely water saturated super critical carbon dioxide is then used to extract the caffeine from the ground coffee in the extraction column. Finally, the coffee oils are returned to the ground, de-caffeinated coffee. Throughout this entire process, the carbon dioxide is always super critical with respect to either temperature or pressure and, during extraction, the carbon dioxide is super critical with respect to both of these. This Specification also discusses the use of liquid carbon dioxide and includes a comparative example showing that, for coffee, approximately eighty times more carbon dioxide is required to be passed through the coffee when the carbon dioxide is in the liquid state than when it is in the super critical state to obtain the required extraction of the coffee oil.

Brief Summary Text (4):
Another British Patent Specification, No. 1,388,581, discusses the use of super critical carbon dioxide as the extracting agent for hops. This Specification mentions that it is possible to obtain an extract from hops using liquid carbon dioxide but again discusses that the dissolving power of liquid carbon dioxide is much lower than that of super critical carbon dioxide. The Specification describes a system in which carbon dioxide gas is compressed until it is super critical with respect to pressure, subsequently heated so that it is super critical with respect to both pressure and temperature, and then passes through an extraction column packed with hops. The super critical carbon dioxide is then cooled so that it is only super critical with respect to pressure and introduced into a separator in which the lower solubility of the matter extracted from the hops in the carbon dioxide which is only super critical with respect to pressure, causes the extracted matter to fall out of solution. The carbon dioxide is then re-cycled through the compressor and extraction chamber. The Specification describes the extract as being a paste varying in colour from light to dark green. The conventional solvent extractions strip out not only the bittering principles but the chlorophyll and presumably the super critical carbon dioxide extracts some of the chlorophyll from the hops. It is very difficult to remove the chlorophyll and other matter from the extract, for example to obtain an isomerised extract, without also losing some of the flavour components.

Brief Summary Text (5):
All of the extraction plants using super critical carbon dioxide work at very high pressures and so result in the plant being very expensive.

Brief Summary Text (6):
We have found that, under carefully controlled conditions of temperature and pressure, liquid carbon dioxide is very selective in its action and it is possible to obtain a golden yellow hop extract which contains the bittering principles of the hops, i.e. the alpha acid fraction of the soft resins contained in the hops together with other required flavour components. The remainder of the extract is composed of inert material that does not influence either the taste or the colour of the extract. This extract is so pure that it can be isomerised directly without requiring any further purification steps. This invention provides a method and plant which enables such an extraction to be carried out very efficiently using liquid carbon dioxide as the extracting agent.

Brief Summary Text (7):
According to a first aspect of this invention, a method of preparing an extract from a material comprises contacting the material to be extracted with carbon dioxide in the liquid state to dissolve the matter to be extracted in liquid carbon dioxide, heating the solution of extract to evaporate carbon dioxide from the solution of extract, compressing and condensing the evaporated carbon dioxide vapour to re-form liquid carbon dioxide and re-cycling the liquid carbon dioxide to contact the material to be extracted, at least part of the heat evolved from the carbon dioxide vapour after its compression and during its change of state from vapour to liquid carbon dioxide being used to cause the change of state of the liquid carbon dioxide in the solution of the extract to evaporate the carbon dioxide from the solution of the extract.

Brief Summary Text (8):
Preferably, the liquid carbon dioxide is subjected to a cooling step before it is

contacted with the material to be extracted.

Brief Summary Text (9):
According to a second aspect of this invention, a plant for the preparation of an extract of hops by extraction with liquid carbon dioxide comprises an extraction chamber having an inlet and an outlet and being arranged to contain the hops to be extracted, a compressor for compressing carbon dioxide gas, a cooler, and a heat exchanger having a primary path for the warmer medium to be cooled and a secondary path for the cooler medium to be warmed, the inlet of the secondary path of the heat exchanger being connected to the outlet of the extraction chamber so that a stream of hop extract dissolved in liquid carbon dioxide emerging from the outlet of the extraction chamber is introduced into the secondary path of the heat exchanger where it receives heat and where carbon dioxide is evaporated, the outlet of the secondary path of the heat exchanger being connected to the compressor so that the carbon dioxide vapour leaving the outlet of the secondary path of the heat exchanger is applied to the compressor where it is compressed, the inlet of the primary path of the heat exchanger being connected to the compressor so that the compressed carbon dioxide vapour warmed by its compression is introduced into the primary path of the heat exchanger where it loses heat to the secondary path and where the carbon dioxide vapour changes state and liquifies to form liquid carbon dioxide, the outlet of the primary path of the heat exchanger being connected to the cooler so that liquid carbon dioxide emerging from the outlet of the primary path of the heat exchanger is further cooled in the cooler and the cooler is connected to the inlet of the extraction chamber so that liquid carbon dioxide is re-cycled to the inlet of the extraction chamber, the secondary path of the heat exchanger including at least one additional outlet for the matter extracted from the hops.

Brief Summary Text (10):
This plant leads to a very efficient extraction being carried out since the plant is in thermal equilibrium, with the sensible heat and latent heat of the compressed carbon dioxide vapour as it is liquified being used to provide the heat required to evaporate carbon dioxide from the solution of the hop extract in the other path of the heat exchanger. The cooling of the liquid carbon dioxide in the cooler before it is introduced into the extraction column reduces the temperature of the liquid carbon dioxide to below its equilibrium temperature corresponding to the operating pressure at that point. This takes the liquid carbon dioxide leaving the heat exchanger off the boil before feeding it to the extraction column so that substantially no carbon dioxide vapour is present in the extraction column which ensures the maximum contact between the liquid carbon dioxide and the hops in the extraction column.

Brief Summary Text (11):
Preferably, the plant includes means to control the flow of coolant through the cooler in dependence upon the temperature of liquid carbon dioxide introduced into the inlet of the extraction chamber to maintain the temperature of the liquid carbon dioxide introduced into the extraction chamber at a constant predetermined temperature. Preferably, the means is arranged to maintain the temperature within a range from 0.degree. to 15.degree. C. It is further preferred that the means is arranged to maintain a temperature of substantially 8.degree. C. This temperature corresponds to the maximum solubility of the alpha acids contained in the hops together with the reasonably low solubility of the beta acids contained in the hops and when the extraction is carried out at this temperature, a particularly beneficial extract is obtained.

Brief Summary Text (12):
Depending upon the ambient temperatures surrounding the plant and the efficiency of its thermal insulation, the plant may be arranged so that the cooler just balances the net input of heat from the compressor but it is preferred that the extraction plant includes a further heat exchanger located between the compressor and the inlet to the primary path of the heat exchanger. Depending upon the ambient temperatures, the degree of thermal insulation, and the exact temperature at which it is required to carry out the extraction and evaporation of the carbon dioxide, this further heat exchanger may add or subtract heat from the compressed carbon dioxide leaving the compressor. This further heat exchanger merely exerts a fine balance on the system.

Brief Summary Text (13):
Preferably, the plant includes means for controlling the flow of heat exchange fluid through the further heat exchanger in dependence upon the pressure subsisting at a point in the plant. In this case, the means preferably senses the pressure subsisting in the secondary path of the heat exchanger and controls the further heat exchanger to maintain the pressure in the secondary path of the heat exchanger at a value such that the temperature in the secondary path of the heat exchanger is within a range from 10.degree. C. to the critical temperature of carbon dioxide. It is preferred that the means maintains the temperature of the secondary path of the heat exchanger within the range from 10.degree. C. to the critical temperature of carbon dioxide, 31.degree. C. since below temperatures of 10.degree. C. solid carbon dioxide hydrate CO.sub.2.8H.sub.2 O is deposited on the heat exchange surfaces of the heat exchanger and this interferes with the efficiency of the heat exchanger.

Brief Summary Text (14):
As carbon dioxide evaporates in the secondary path of the heat exchanger and consequently the concentration of the matter extracted from the hops increases, the concentration gradually exceeds its solubility in liquid carbon dioxide and so the extract is precipitated from solution. The hop extract has the form of a yellow mobile liquid and forms a separate layer in the lower parts of the secondary path of the heat exchanger.

Brief Summary Text (15):
Preferably, the primary path of the heat exchanger also includes a vent outlet at its uppermost end. This vent outlet enables any volatile impurities present in the system to be vented. This is particularly important in the case of oxygen since it is difficult to ensure that all the air is purged from the hops by flushing with carbon dioxide gas and, in the case of inefficient flushing of the system with carbon dioxide, air entrained in the hops leads to the presence of air in the system during extraction. Nitrogen is inert and has no real effect on the system but oxygen attacks the extracted material and causes oxidation. The provision of the vent in the primary path from the heat exchanger together with the change of state which takes place in the primary path of the heat exchanger enables the volatile impurities to be vented from the system resulting in substantially no oxidation of the matter extracted from the hops. This is a considerable advantage over the systems using super critical carbon dioxide to extract matter since, with a system in which carbon dioxide is always super critical in respect to pressure, temperature or both, it is impossible to vent any volatile impurities which are present in the system.

Brief Summary Text (16):
At present in the brewing industry, a process of "dry hopping" is carried out in which hops are steeped in the fermented wort to increase the concentration of hop oils in the wort and improve the taste and bouquet of the completed beer. We have found that the hop oils can be isolated separately from the bittering principles of the hops and that the proportion of the hop oils contained in the extracted matter can also be varied. The concentration of hop oils in the liquid carbon dioxide in the secondary path of the heat exchanger gradually builds up and, whilst some hop oils are mixed with the alpha and beta acid fraction of the soft resins, a far greater quantity remain in solution with the liquid carbon dioxide.

Brief Summary Text (17):
To recover a different product from the secondary path of the heat exchanger a further outlet may be provided in communication with the solution of extract in liquid carbon dioxide in the secondary path of the heat exchanger and the plant may also include an evaporator arranged to receive the solution of the extract and to evaporate carbon dioxide to leave a product which is rich in hop oils. The carbon dioxide evaporated from the evaporator is returned to the compressor and again re-cycled through the system. This alternative product has a high proportion of hop oils, of the order or 50%, as well as some alpha and beta acids. Such a hop extract would give far more flavour and bouquet to a beer and the two different products may be blended to give the required proportions of bittering agents and flavour components.

Brief Summary Text (18):

The plant may also include a distillation column in series with the outlet of the secondary path of the heat exchanger and the inlet to the compressor. With a distillation column in this position, a pure extract of hop oils can be obtained. The hop oils may be separated from any carbon dioxide or it may remain as a solution in liquid carbon dioxide so that they can be injected into the brewed beer during its carbonation at the completion of the brewing process in a somewhat analogous fashion to the existing "dry hopping" process.

Brief Summary Text (19):
Finally, the plant may also include means to introduce a further component into the secondary path of the heat exchanger. This further component which is usually an organic solvent and preferably ethanol, can be injected into the secondary path of the heat exchanger and act as an anti-freeze to prevent the formation of the solid carbon dioxide hydrate CO.sub.2.8H.sub.2 O and to allow the secondary path of the heat exchanger to operate at substantially lower temperatures, or alternatively this additional component may be used to change the equilibrium occurring in the secondary path of the heat exchanger. In this way, an additional component, particularly when it is ethanol, can produce an homogeneous extract having a high proportion of hop oils in combination with the alpha and beta acids and the matter extracted from the hops may have the form of a tincture of the extract in ethanol. The addition of this further component into the system in the secondary path of the heat exchanger can be used to cause separation of the various phases in the secondary path of the heat exchanger and to modify the volatility of the various components to enhance or depress the concentration of hop oils leaving the outlet of the secondary path of the heat exchanger.

Detailed Description Text (1):
The basic plant comprises two similar extraction chambers 1 and 2 arranged in parallel, with a pair of isolating valves 3 and 4 at opposite ends of each chamber 1 and 2 respectively. Each chamber is formed as a column and includes a filter plate covering its inlet and outlet to prevent the hops from leaving the chambers but to allow liquid carbon dioxide to percolate up through the hops in each chamber. The outlets at the tops of the chambers 1 and 2 are connected through a pipe 5 to a flow control valve 6. The outlet of the flow control valve 6 leads into a secondary path 7 of a heat exchanger 8. The flow control valve 6 is controlled by a liquid level sensor 9 so that a substantially constant level is maintained in the secondary path 7 of the heat exchanger 8.

Detailed Description Text (2):
A pipe 10 leads from an outlet of the secondary path 7 of the heat exchanger 8 to the inlet of a compressor 11. The outlet of the compressor 11 is connected to a heat exchanger 12 which, in this example, is a cooler, and the outlet of the cooler is connected to a primary path 13 of the heat exchanger 8. An outlet of the primary path 13 of the heat exchanger 8 is connected to a liquid receiver vessel 14 and an outlet from the liquid receiver vessel 14 is connected through an isolating valve 15 to an inlet of a sub-cooler 16. The outlet of the sub-cooler 16 leads directly into the inlet of the extraction chambers 1 and 2 through the valves 3 and 4. The quantity of coolant passing through the sub-cooler 16 is controlled by a flow control valve 17 under the control of a temperature sensor 18 which detects the temperature of the liquid carbon dioxide leaving the sub-cooler 16 and varies the flow of coolant through the sub-cooler 16 to maintain a constant temperature at the output of the sub-cooler 16. The flow of coolant through the heat exchanger 12 is controlled by a flow control valve 19 under the control of a pressure sensor 20. The heat exchanger 12 serves to trim the overall heat balance in the system and, as the pressure within the system increases, the flow control valve 19 is opened to increase the degree of cooling that occurs in the heat exchanger 12.

Detailed Description Text (3):
The secondary path 7 of the heat exchanger 8 includes a further vent outlet 21 controlled by a flow control valve 22 and a product outlet 23 controlled by a flow control valve 24. A vent outlet enables volatile material to be vented from the secondary path 7 of the heat exchanger 8 and the product outlet 23 allows the extracted matter to be removed from the secondary path of the heat exchanger 8. The liquid receiver 14 includes a further inlet 25 under the control of a flow control valve 26 through which carbon dioxide is introduced into the system to fill the

system initially and to make up any losses.

Detailed Description Text (4):
The entire plant is made from stainless steel and the plant is arranged to operate at pressures of the order of 750 p.s.i.g. Since the heat exchanger 8 has carbon dioxide in both its primary and secondary paths the differential pressure between the primary and secondary path of the heat exchanger 8 is merely that generated by the compressor 11 and this differential pressure is quite low, of the order of 100 p.s.i.g. This small differential pressure between the primary and secondary paths of the heat exchanger simplifies its construction.

Detailed Description Text (5):
With the valves 3 closed to isolate the extraction chamber 1, the chamber 1 is packed with milled hops. Most of the air is purged from the chamber 1 using gaseous carbon dioxide and then the valves 3 are opened to pressurize the chamber 1 and allow liquid carbon dioxide to percolate up through the hops in the chamber 1. As the liquid carbon dioxide percolates through the extraction chamber 1 it extracts the parts of the hops which are soluble in liquid carbon dioxide. Principally, the matter extracted from the hops are the hop oils and the alpha and beta acid fraction of the soft resins contained in the hops, the alpha acids forming the bittering principles of the hops, and the hop oils forming the main flavour and aroma components of the hops. The solution of the extracted matter passes along the pipe 5 and, through the control valve 6 into the secondary path 7 of the heat exchanger 8. In the heat exchanger 8 the solution is warmed causing a change of state of the carbon dioxide to occur and the carbon dioxide vapour boiled off from the solution then leaves the secondary path 7 of the heat exchanger 8 through the pipe 10 to the compressor 11. The carbon dioxide vapour is compressed in the compressor 11 and, during compression, the vapour is heated on account of the work done in compressing the gas. The compressed gas passes through the heat exchanger 12 and then to the primary path 13 of the heat exchanger 8. In this primary path, the compressed gas changes its state and forms liquid carbon dioxide. The heat removed from the compressed gas as it is initially cooled to the condensation temperature and then the latent heat evolved as the carbon dioxide changes its state from the vapour to the liquid phase is transferred to the solution of extract in the secondary path 7 of the heat exchanger 8. This leads to boiling of the solution to change the state of the liquid carbon dioxide in the secondary path 7 of the heat exchanger 8 to evolve further carbon dioxide vapour from the solution in the secondary path 7. The liquid carbon dioxide formed in the primary path 13 of the heat exchanger 8 passes into the liquid receiver 14 where any carbon dioxide vapour entrained with the liquid carbon dioxide is separated. The liquid carbon dioxide from the liquid receiver 14 then passes through a flow control valve 15 to the sub-cooler 16 where it is cooled to a temperature below its equilibrium temperature at the pressure subsisting in the sub-cooler 16. It has been found that if the liquid carbon dioxide is cooled to a temperature of 8.degree. C. a particularly beneficial extraction occurs in the chambers 1 and 2. Therefore, the temperature sensor 18 controls the flow of coolant through the sub-cooler 16 via the flow control valve 17 to ensure that the liquid carbon dioxide leaves the sub-cooler 16 at a temperature of 8.degree. C. This liquid carbon dioxide at the temperature of 8.degree. C. is then re-cycled through the extraction chamber 1 and thence through the remainder of the system.

Detailed Description Text (6):
Once all the matter has been extracted from the hops in the extraction chamber 1, the flow control valves 6 and 15 are closed and the liquid carbon dioxide remaining in the extraction chamber 1 is used to purge air from the extraction chamber 2 and also to partly pressurize the extraction chamber 2. Valves 3 are then closed to isolate the spent hops in the extraction chamber 1 and the flow control valves 6 and 15 are once again opened to allow extraction to continue only this time through the hops contained in the extraction chamber 2. Whilst the hops in the extraction chamber 2 are being extracted the spent hops in extraction chamber 1 are removed from the chamber 1 and the chamber is re-packed with fresh hops.

Detailed Description Text (7):
The heat exchanger 12 is used to trim the thermal equilibrium of the entire system. In general, the heat required for vaporising the liquid carbon dioxide from the

solution of extract in the secondary path 7 of the heat exchanger 8 is more or less balanced by the heat evolved on the change of state of the gaseous carbon dioxide in the primary path 13 into liquid carbon dioxide; with the sub-cooler 16 just about balancing the heat added to the system by the work done on the gas in the compressor 11. However, depending on the ambient temperature surrounding the plant and the efficiency of its thermal insulation the flow of coolant through the heat exchanger 12 is used to absorb the excess heat in the system. The flow of coolant through the heat exchanger 12 is controlled via the flow control valve 19 from a pressure controller which senses the pressure subsisting in the secondary path 7 of the heat exchanger 8. It has been found that if the pressure subsisting in the secondary path 7 of the heat exchanger 8 is controlled so that a temperature of just above 10.degree. C. occurs in the secondary path of the heat exchanger 7 the plant operates in a very satisfactory manner since carbon dioxide hydrate CO.sub.2.8H.sub.2 O is not formed on the heat exchange surfaces of the secondary path of the heat exchanger 8 to impede the transfer of heat between the primary and secondary paths of the heat exchanger 8.

Detailed Description Text (8):
As the carbon dioxide is evaporated from the secondary path 7 of the heat exchanger 8, the concentration of the matter extracted from the hops, particularly the concentration of the alpha and beta acids, builds up in the secondary path of the heat exchanger and the concentration of the alpha and beta acids exceeds their solubility in liquid carbon dioxide. After this saturation point has been reached the alpha and beta acids are precipitated from solution and form a separate mobile layer in the lowermost end of the secondary path 7 of the heat exchanger 8. This separate phase containing the alpha and beta acids, and some of the hop oils, is drawn off the secondary path 7 of the heat exchanger 8 by opening the flow control valve 24 to allow the product to leave the secondary path 7 of the heat exchanger 8 out of the outlet 23.

Detailed Description Text (9):
Any air remaining with the hops in the extraction chambers 1 or 2 after the initial flushing operation with carbon dioxide tends to build-up in the secondary path 7 of the heat exchanger 8. This build-up of air, particularly the oxygen contained in the air, leads to oxidative changes in the matter extracted from the hops which impairs the quality of the extract. Accordingly, volatile impurities in the secondary path of the heat exchanger are vented through the outlet 21 under the control of the flow control valve 22. This enables all of the air and other gaseous impurities present in the system to be vented and removed from the system and ensures that a top quality extract is obtained.

Detailed Description Text (10):
As the carbon dioxide vapour is evaporated from the secondary path 7 of the heat exchanger 8 the concentration of hop oils in the liquid carbon dioxide in the secondary path 7 increases. These hop oils are a valuable part of the matter extracted from the hops since they contain a large proportion of the flavour and aroma principles of the hops. A first modification of the plant shown in FIG. 2 enables an alternative extract to be obtained which includes a higher percentage of hop oils. This plant is identical to the basic plant except that the secondary path 7 of the heat exchanger 8 includes a further outlet 27 controlled by a flow control valve 28 and this further outlet leads to an evaporator 29. The top of the evaporator is connected through a flow control valve 30 to the pipe and the base of the evaporator includes an outlet 31 controlled by a flow control valve 32.

Detailed Description Text (11):
The operation of this modified plant is basically similar and a hop extract containing mainly the alpha and beta acids is withdrawn from the outlet 23 through the flow control valve 24. However, in addition, the liquid carbon dioxide containing the solution of the extract is also removed from the secondary path 7 of the heat exchanger 8 through the outlet 27 under the control of the flow control valve 28. This solution is then evaporated in the evaporator 29 and the carbon dioxide boiled off is returned to the pipe 10 and thence the compressor 11 and re-cycled through the plant. The residue remaining in the evaporator 29 is drawn off the base through the outlet 31 and this residue contains some alpha and beta acids but is rich in hop oils, for example it contains up to 50% of hop oils. The two

different extracts may be mixed to enhance the proportion of hop oils or this further extract which is high in hop oils may be used alone.

Detailed Description Text (12):
A second modification of the basic plant is shown in FIG. 3 and this modification enables the hop oils to be isolated so that the product of this plant will be a hop extract containing principally the alpha and beta acids together with approximately 10% of hop oils, and a pure hop oil extract or, a concentrated solution of hop oils in liquid carbon dioxide. In this second modification, a distillation tower 33 is connected in parallel with the pipe 10 and valves 34, 35 and 36 are arranged so that the distillation tower 33 can be connected in series with the outlet from the secondary path of the heat exchanger 8 and the compressor 11. A further pipe 37 and flow control valves 38 and 39 supply the distillation tower 33 with liquid carbon dioxide. As the concentration of hop oils in the carbon dioxide in the secondary path 7 of the heat exchanger 8 builds up, a point is reached where hop oils will be carried off with the evaporated carbon dioxide. To recover the hop oils, the valve 35 is closed and the valves 34 and 36 opened and this mixed vapour containing carbon dioxide and the hop oils is passed through the distillation tower 33. Liquid carbon dioxide from the liquid receiver 14 is supplied to the top of the distillation tower 33 and scrubs the vapour to remove the hop oils from the vapour phase. Carbon dioxide is then boiled off from the base of the distillation tower 33 to leave a product which consists exclusively of hop oils or a concentrated solution of hop oils in liquid carbon dioxide. The hop oils can be injected into already brewed beer to improve the flavour and bouquet of the beer and, when the beer is to be subjected to a final carbonation step it is preferred for the hop oils to be injected into the brewed beer in solution with liquid carbon dioxide. The quantity of hop oils carried over into the vapour phase of the carbon dioxide leaving the secondary path of the heat exchanger 8 can be increased by operating the plant with the secondary path of the heat exchanger 8 close to the critical temperature of carbon dioxide which is about 31.degree. C. The reason for this is that the carry-over of the hop oils depends upon the relative solubility of the hop oils in the liquid and vapour phases of the carbon dioxide and the solubility of the hop oils in the carbon dioxide vapour at a temperature just above the critical temperature is substantially the same as the solubility of the hop oils in the carbon dioxide liquid at just below the critical temperature.

Detailed Description Text (13):
In a final modification of the basic plant shown in FIG. 4, a solvent is injected into the secondary path 7 of the heat exchanger 8. Once again the plant is generally similar to the basic plant except that the secondary path 7 of the heat exchanger 8 includes a further inlet 40 controlled by a flow control valve 41 and a container 42 containing the solvent is connected to the valve 41 through a pump 43. The preferred solvent is ethanol and the injection of ethanol into the secondary path of the heat exchanger firstly acts as an anti-freeze and so prevents the solid carbon dioxide hydrate CO.sub.2.8H.sub.2 O from being formed and being deposited on the walls of the secondary path of the heat exchanger and this enables the temperature in the secondary path of the heat exchanger to be less than 10.degree. C. Further, if a greater quantity of ethanol is injected into the secondary path of the heat exchanger, the ethanol will homogenise the two separate phases in the secondary path of the heat exchanger to form a single phase and then this single phase can be drawn off and the carbon dioxide evaporated from the single phase. After the carbon dioxide has been evaporated an extract will remain which will have the form of a tincture of hop extract in ethanol. The ratio of the bittering principles to the hop oils of this resulting extract contained in this tincture will be the same as the ratio of the bittering principles to the hop oils contained in the hops. This modification shown in FIG. 4 may also be included together with the modifications shown in FIG. 2 or FIG. 3 and then the addition of an additional component into the system present in the secondary path of the heat exchanger 8 may be used to influence the volatility of the system in the secondary path 7 of the heat exchanger 8 and, in this way, enhance or depress the carry over of the particular components from the liquid to the vapour phase.

CLAIMS:

1. A plant for the preparation of an extract of hops by extraction with liquid

carbon dioxide, said plant comprising an extraction chamber arranged to contain hops to be extracted, an inlet and an outlet of said extraction chamber; a compressor for compressing carbon dioxide gas; a cooler; a heat exchanger including a primary path for a warmer medium to be cooled said primary path having an inlet and an outlet, and a secondary path for a cooler medium to be warmed, said secondary path having an inlet and an outlet; means connecting said inlet of said secondary path of said heat exchanger to said outlet of said extraction chamber whereby a stream of hop extract dissolved in liquid carbon dioxide emerging from said outlet of said extraction chamber is introduced into said secondary path of said heat exchanger wherein said stream receives heat and wherein carbon dioxide is evaporated; means connecting said outlet of said secondary path of said heat exchanger to said compressor whereby said carbon dioxide vapour leaving said outlet of said secondary path of said heat exchanger is applied to said compressor wherein said vapour is compressed; means connecting said inlet of said primary path of said heat exchanger to said compressor whereby said compressed carbon dioxide vapour warmed by its compression is introduced into said primary path of said heat exchanger wherein it loses heat to said secondary path and wherein said carbon dioxide vapour changes state and liquifies to re-form liquid carbon dioxide; means connecting said outlet of said primary path of said heat exchanger to said cooler whereby liquid carbon dioxide emerging from said outlet of said primary path of said heat exchanger is further cooled in said cooler; means connecting said cooler to said inlet of said extraction chamber whereby re-formed liquid carbon dioxide is re-cycled to said inlet of said extraction chamber; and, an additional outlet in said secondary path of said heat exchanger for said matter extracted from said hops.

2. The plant of claim 1, wherein means to control the flow of coolant through said cooler in dependence upon the temperature of liquid carbon dioxide introduced into said inlet of said extraction chamber are provided whereby said temperature of said liquid carbon dioxide is maintained at a constant predetermined temperature.

6. The plant of claim 5, wherein said means for controlling said flow of heat exchange fluid through said further heat exchanger include sensor means for sensing the pressure subsisting in said secondary path of said heat exchanger, and control means for controlling the flow through said further heat exchanger to maintain said pressure in said secondary path of said heat exchanger at a value whereby said temperature in said secondary path of said heat exchanger is within a range from 10.degree. C. to the critical temperature of carbon dioxide.

8. The plant of claim 1, wherein said secondary path of said heat exchanger includes a further outlet, said further outlet communicating with said solution of extract in liquid carbon dioxide in said secondary path of said heat exchanger, and wherein said plant includes an evaporator, and means connecting said further outlet to said evaporator whereby said evaporator receives said solution of extract and evaporates carbon dioxide to leave a product rich in hop oils.

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L9: Entry 34 of 35 File: DWPI Jul 10, 1982

DERWENT-ACC-NO: 1982-62933E
DERWENT-WEEK: 198230

TITLE: Addn. of hop extracts to beer using liq. carbon di:oxide as solvent - by in-line mixing of the extract into liq. carbon di:oxide as it is dosed in-line to the beer

PATENT-ASSIGNEE:

ASSIGNEE	CODE
BAKER C D	BAKEI

PRIORITY-DATA: 1982RD-0219046 (June 20, 1982)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
RD 219046 A	July 10, 1982		000	

INT-CL (IPC): C12C 0/01

ABSTRACTED-PUB-NO: RD 219046A
BASIC-ABSTRACT:

Process is described for use of liq. CO2 as a solvent for the addn. and dispersal of liq. CO2 extracts of hops added to beer as a replacement for dry hopping. It comprises in-line mixing of the hop extract into liquid carbon dioxide as it is itself dosed in-line to the beer.

If adequate mixing of the hop extract (if necessary dissolved in a small amt. of ethanol depending upon its initial viscosity) and liquid carbon dioxide can be obtd. either by an in-line mixing device, or by using an appropriate nozzle at the injection point of the liquid carbon dioxide soln. into the beer, no high pressure vessel other than that for the storage of liq. carbon dioxide will be required.

Provided that a constant pressure can be maintained in the liq. carbon dioxide vessel (for example by using an appropriate relief valve) to ensure constant flow of CO2 into the beer, the only pressure pump required will be for the hop oil, for which readily available high pressure liquid chromatography pumps would be suitable for beer flow rates up to 200 barrels per hr.

TITLE-TERMS: ADD HOP EXTRACT BEER LIQUID CARBON DI OXIDE SOLVENT LINE MIX EXTRACT LIQUID CARBON DI OXIDE DOSE LINE BEER

DERWENT-CLASS: D16

CPI-CODES: D05-B;

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L5: Entry 13 of 39 File: USPT Dec 15, 1992

DOCUMENT-IDENTIFIER: US 5170727 A
TITLE: Supercritical fluids as diluents in combustion of liquid fuels and waste materials

Brief Summary Text (38):
For food and pharmaceutical applications, supercritical carbon dioxide is the most prominent supercritical fluid utilized. In addition to aforementioned extractions in decaffeination and denicotinization processes, other processes include acids from hops, extraction of oils from soybean flake and corn germ in which, in addition to carbon dioxide, ethane, propane, and nitrous oxide are used.

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L5: Entry 24 of 39 File: USPT Sep 8, 1987

DOCUMENT-IDENTIFIER: US 4692280 A
TITLE: Purification of fish oils

Brief Summary Text (10):
Supercritical carbon dioxide, which is carbon dioxide under high pressure above its critical temperature of 31.degree. C., i.e., carbon dioxide gas non-liquefiable under pressure, is known to have selective solvent properties for the preparation of human food-grade products. For example, U.S. Pat. No. 4,495,207 describes its use in extracting lipids from corn germ. Supercritical carbon dioxide is used commercially for the removal of caffeine from coffee and in the extraction of the essence of hops for use in the brewing of beer. The fractionation of fish oil esters using supercritical carbon dioxide is described by Eisenbach, Ber. Bunsenges, Phys. Chem., 88, 882-887 (1984) and in commonly assigned copending application Ser. No. 879,543, filed June 24, 1986.

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L5: Entry 25 of 39 File: USPT Mar 3, 1987

DOCUMENT-IDENTIFIER: US 4647464 A
TITLE: Hop flavor constituents adsorbed on fumed silicon dioxide

Brief Summary Text (4):
Accordingly, efforts to overcome these control problems have been made in recent years by carefully extracting hops aromas with carbon dioxide, by making emulsions of these oils and aroma using Polysorbate 80.TM. (polyoxyethylene (20) sorbitan mono-oleate), and also by adding the oils as a portion of isohumulones as described as an option in U.S. Pat. No. 3,486,906 (Todd). Each of these efforts has its shortcoming. The Polysorbate emulsions introduce Polysorbate into the beer, which can become rancid and objectionable in such a delicately flavored system as well as potentially requiring label disclosure. The isohumulone emulsions may not be stable, and the hop oil may float out, so that it does not dissolve in the beer. Gum emulsions present the same potential difficulty and uncertainty. So do dispersions of hop oil on a water-soluble substrate, such as dextrose. It is well known to the art that spice flavorings may be dispersed on dextrose, and the dispersions added to a food such as sausage as a flavoring. Such a system has never been practical for hop flavors, since the oils and bitter acids tend to float out in the beer, and are not uniformly dissolved. Thus both efficiency is lost, and objectionable "hot spots" of flavor may be present.

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L9: Entry 31 of 35 File: USPT Aug 1, 1978

DOCUMENT-IDENTIFIER: US 4104409 A
TITLE: Production of hop extracts

Abstract Text (1):
Process for extraction of hops utilizing supercritical gases such as carbon dioxide.

Brief Summary Text (9):
A process has now been discovered for the production of hop extracts in which air dried hops are extracted with extraction solvents in the supercritical state with respect to temperature and pressure. Suitable solvents include carbon dioxide, saturated and unsaturated hydrocarbons containing up to about three carbon atoms; preferably those in which at least fifty percent of the hydrogen atoms of the parent hydrocarbon are replaced with halogen; sulfur hexafluoride; nitrous oxide and mixtures of these having a critical temperature of from about 30.degree. to 100.degree. C. The extracts are separated from the solvent gas by reducing the pressure or temperature or both to below the critical points.

Brief Summary Text (10):
Carbon dioxide is the most preferred extraction medium, but others such as SF.sub.6, CHF.sub.3, CF.sub.3 Cl, CF.sub.2 .dbd. CH.sub.2, CHF.sub.2 Cl, C.sub.3 F.sub.8, CH.sub.2 .dbd.CH.sub.2, CH.sub.3 CH.sub.3 and N.sub.2 O either singly, mixed with each other or with carbon dioxide can be employed. Supercritical CO.sub.2 in the sense of this invention is CO.sub.2 the temperature of which is above the critical temperature of CO.sub.2 (31.3.degree. C), while its pressure is above the critical pressure (about 73 atmospheres). Other supercritical gases used in the invention may be similarly described. Typically, the critical pressure will be at least 70 atmospheres and the temperature at least 30.degree. C.

Detailed Description Text (1):
For illustrative purposes, the invention will be principally described with reference to the most preferred solvent carbon dioxide. The presently preferred process will be best understood by reference to FIG. 1.

Detailed Description Text (2):
The gas used in the process is stored in tank 1 controlled by valve 2. In carrying out the method of the invention, the entire system is flushed with dry CO.sub.2 and valves 3, 4, 5 and 6 are opened. All other valves are closed. Liquid carbon dioxide flows from tank 1 to liquid gas pump 11 and then into heat exchanger 12 where it is brought to supercritical conditions of temperature and pressure. The resulting gas flows through valve 7 which is opened and into pressure pipe 13 which is formed with heat jacket and equipped with filter plates having a filter threshold of five millimicrons to support the hops.

Detailed Description Text (14):
In another modification of the method, the hops liberated of the total resin and the essential oils by the treatment with dry, supercritical CO.sub.2, cannot be treated directly with water, but first in a second step with wet, supercritical CO.sub.2. The wetting of the gas current is effected in tank 19, which is cut into the circuit through valves 8 and 9. There is thus obtained an extract fraction whose constituents have some tannin character. The water extract thus obtained, which is either dry or hydrous, as desired, is then added to the extract obtained in the

first step and can then be used either alone or mixed with the extract portion obtained from the strictly aqueous extraction (third step), which can be either spray- or freeze-dried.

CLAIMS:

4. A process as in claim 1 wherein the extraction gas is carbon dioxide.

5. A process according to claim 1 in which the extraction solvent gas is dry carbon dioxide and resin and essential oils are thereby extracted from the hops in a first extraction step, and the extracted hops are further extracted with wet supercritical carbon dioxide, in a second extraction step whereby tannin is extracted from the hops.

13. A process according to claim 1 wherein the extraction gas is selected from the group consisting of carbon dioxide; saturated and unsaturated hydrocarbons containing up to about three carbon atoms; nitrous oxide; saturated and unsaturated halogenated hydrocarbons containing up to about three carbon atoms; and mixtures of these.

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L5: Entry 10 of 39 File: USPT May 7, 1996

DOCUMENT-IDENTIFIER: US 5514401 A
TITLE: Supercritical fluid extraction of cholesterol from liquid egg yolk

Brief Summary Text (14):
In recent years, supercritical fluid extraction (SFE) has become an acceptable method for coffee and tea decaffeination, hops extraction, and flavor and spice extract production. Recent research indicates that many other additional commercial applications are likely to become utilized in food industry since the primarily used solvent for supercritical extraction, namely carbon dioxide, is safe, nontoxic, non-reactive, inexpensive, and leaves no residue in the food. The supercritical extraction technology thus provides safer and more economic alternative to produce healthier higher quality foods.

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L5: Entry 11 of 39 File: USPT Feb 15, 1994

DOCUMENT-IDENTIFIER: US 5286506 A
TITLE: Inhibition of food pathogens by hop acids

Brief Summary Text (8):
One of the historical roles of hops in beer making, namely the bacteriostatic function, has been made obsolete in the modern manufacture of beer by aseptic fermentation and packaging. The bitter acids component of the hops and particularly the beta-acids have now been found to be unexpectedly useful as bacteriocides in food products. The most prevalent groups of bitter acids found as components of hops are the alpha-acids and the beta-acids, also referred to as humulones and lupulones, respectively. Both contribute bitterness to beer, but the alpha-acids are much more intense in this regard than the beta-acids. Producers of hop extracts have thus recently developed a technique to separate the two acid fractions using liquid carbon dioxide under supercritical conditions. A by-product of the operation is a product which contains approximately 61 weight percent beta-acids, the remainder consisting essentially of hop resins.

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L5: Entry 12 of 39 File: USPT Nov 23, 1993

DOCUMENT-IDENTIFIER: US 5264236 A
TITLE: Method for production of hop extracts and hop extracts obtained thereby

Detailed Description Text (6):
The subcritical or supercritical carbon dioxide used as extracting agent in the present invention is non-flammable, harmless, cheap, and easy to handle, with a critical temperature of 31.3.degree. C. and a critical pressure of 72.9 atm. In addition, a supercritical fluid has a density close to that of liquid and a high diffusion coefficient, close to that of a gas, which characteristics give the supercritical fluid the capability of quickly extracting large amounts of various compounds with high yield. Moreover, supercritical carbon dioxide is easily separated from the extracts by slightly changing the pressure or temperature, and even offers a bacteriostatic or bactericidal effect, as an advantageous characteristic of carbon dioxide. It is thus harmless to humans, and sanitary. For these reasons, it is especially suitable for use in foods and pharmaceuticals, and is preferably used to obtain hop extracts, the desired product of the present invention.

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USPT,PGPB,JPAB,EPAB,DWPI,TDBD	humulone	195	L1

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S5347	U	USPT,PGPB,JPAB,EPAB,DWPI,TDBD	(co2 or carbon dioxide) same ((hop or hops)same (capsule or tablet or injection or inject or inject$6 or spray or lotion or food))	2003-01-23 16:46:31	
S5346	U	USPT,PGPB,JPAB,EPAB,DWPI,TDBD	co2 or carbon dioxide	2003-01-23 16:46:15	
S5345	U	USPT,PGPB,JPAB,EPAB,DWPI,TDBD	(hop or hops) same (capsule or tablet or injection or inject or inject$6 or spray or lotion or food)	2003-01-23 16:45:55	
S5344	U	USPT,PGPB,JPAB,EPAB,DWPI,TDBD	capsule or tablet or injection or inject or inject$6 or spray or lotion or food	2003-01-23 16:39:05	
S5343	U	USPT,PGPB,JPAB,EPAB,DWPI,TDBD	hop or hops	2003-01-23 16:38:13	
S5342	U	USPT,PGPB,JPAB,EPAB,DWPI,TDBD	((noni or morinda citrifolia)and (juice))	2003-01-23 16:37:59	
S5341	U	USPT,PGPB,JPAB,EPAB,DWPI,TDBD	(noni or morinda citrifolia) and (juice)	2003-01-22 14:16:14	
S5340	U	USPT,PGPB,JPAB,EPAB,DWPI,TDBD	juice	2003-01-22 14:15:43	
S5339	U	USPT,PGPB,JPAB,EPAB,DWPI,TDBD	noni or morinda citrifolia	2003-01-22 14:15:25	

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L4: Entry 334 of 334 File: DWPI

DERWENT-ACC-NO: 1966-38536F
DERWENT-WEEK: 196800

TITLE: Hormonal component hop extract

PATENT-ASSIGNEE:

ASSIGNEE	CODE
COSMETOLOGY RES INST	COSY

PRIORITY-DATA: 1967SU-1152128 (April 28, 1967)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
SU 219112 A			000	

ABSTRACTED-PUB-NO: SU 219112A
BASIC-ABSTRACT:

Hormonal component useful in formulating cosmetic prepns. such as skin creams, lotions and shampoos is a hop extract possessing an estrogenic activity of 340-1700 I.U./g. The extract was found to be slightly toxic and non-irritatory. Cosmetic formulations comprising it can be used for prevention and treatment of various skin affections.

TITLE-TERMS: HORMONE COMPONENT HOP EXTRACT

DERWENT-CLASS: B00

CPI-CODES: B04-A07F; B12-A07; B12-G04;

CHEMICAL-CODES:

Chemical Indexing M0 *01*
Fragmentation Code
V400 V406 N160 P940 P620 R000 M423 M900

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L4: Entry 318 of 334 File: DWPI May 11, 1978

DERWENT-ACC-NO: 1978-35230A
DERWENT-WEEK: 197820

TITLE: Bacteriostatic deodorant compsn., e.g. soap, contg. hop extract - which is opt. free of humulone and is used for eliminating or masking body odour

INVENTOR: OWADES, J L

PATENT-ASSIGNEE:

ASSIGNEE	CODE
STEINER S S INC	STEIN

PRIORITY-DATA: 1976US-0739305 (November 5, 1976)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
DE 2749274 A	May 11, 1978		000	
CA 1112182 A	November 10, 1981		000	

INT-CL (IPC): A61K 7/32

ABSTRACTED-PUB-NO: DE 2749274A
BASIC-ABSTRACT:

Bacteriostatic deodorant compsns. for body hygiene contains >=1 (1-5) wt.% bactericidal hop extract, as active ingredient for eliminating or masking body odour caused by Gram positive sweat-decomposing bacteria.

The deodorant can be used as soap or stick (preferred), or a liquid, lotion, atomised liquid, cream, powder or oil. Body odour-causing bacteria, e.g. Staphylococcus aureus and Staphylococcus epidermis, are effectively inhibited on using compsns. contg. as little as 1% hop extract. The hop extract is non-toxic, miscible with other cosmetic ingredients and does not irritate the skin.

TITLE-TERMS: BACTERIA DEODORISE COMPOSITION SOAP CONTAIN HOP EXTRACT OPTION FREE HUMULONE ELIMINATE MASK BODY ODOUR

DERWENT-CLASS: D22

CPI-CODES: D08-B09;

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L7: Entry 30 of 44 File: USPT Aug 19, 1980

DOCUMENT-IDENTIFIER: US 4218491 A
TITLE: Hop extraction with carbon dioxide

Abstract Text (1):
A method of preparing a hop extract is described in which liquid carbon dioxide is passed at a temperature of from -5.degree. C. to +15.degree. C. through a column of hop material to extract .alpha.-acids contained therein. The method gives a primary extract which is of much higher purity than has been previously described particularly in comparison to presently commercially available solvent extracts.

Brief Summary Text (7):
Fluid carbon dioxide either in the form of the liquid or the supercritical gas has been suggested as an extraction medium for hops. Thus, British Patent Specification No. 1388581 describes a method of making a hop extract by extracting hops with a variety of gases in the supercritical state with respect to temperature and pressure. Carbon dioxide is stated to be the most preferred gas. Extraction under such supercritical conditions with CO.sub.2 typically yields an olive-green pasty product which contains .alpha.-acids, .beta.-acids, uncharacterized soft resins, hard resins and small quantities of tannins. The extraction conditions can be varied to give yields of .alpha.-acids proportionately higher than the concentrations in the starting hops; the best extracts described, however, containing only about one-third .alpha.-acids. The optimum extraction conditions are stated to involve extraction under a pressure substantially in excess of the critical pressure, which for CO.sub.2 is about 72.8 atmospheres, preferably in excess of 100 atmospheres (gauge) and temperatures of from 40.degree. to 50.degree. C.

Brief Summary Text (11):
The present invention accordingly provides a method of making a hop extract (of high purity) which method comprises passing liquid carbon dioxide through a column of hop material at a temperature of from -5.degree. C. to +15.degree. C. to extract at least a portion of the .alpha.-acids contained in the hops into the liquid carbon dioxide and subsequently recovering a hop extract of high purity from the liquid carbon dioxide.

Brief Summary Text (25):
A factor which is very important practically relates to the pressure under which the extraction is done. Clearly for the carbon dioxide to remain in a liquid, the pressure in the system cannot be less than the vapour pressure of CO.sub.2 at the temperature within the system. Thus, the higher the temperature at which the extraction is performed the higher the pressure that the extractor must be capable of withstanding. The capital cost and complexity of and the difficulty in maintaining pressure equipment of the kind used in performing the method of extraction of the invention is directly related to the pressures under which it has to operate and it is thus advantageous to operate at sub-ambient temperatures in order to take advantage of the possible reduction of operating pressure. Operating at sub-ambient temperatures involves the use of refrigeration equipment and thermal insulation which is not necessary for operation at ambient temperature. The lower the extraction temperature the more expensive is this additional equipment. The relationship of vapour pressure and the solubilities of .alpha.-acids and .beta.-acids as functions of temperature are illustrated in FIG. 1.

Brief Summary Text (26):

The outside limits on the temperature of extraction in the present invention are -5.degree. C. and +15.degree. C. The preferred temperature depends on the particular technique adopted and in particular for the isolation of the extract. We envisage that isolation will be achieved by evaporating or boiling off the liquid CO.sub.2. This evaporation is preferably done under approximately constant pressure conditions to avoid difficulties associated with freezing which can arise if the pressure is simply released to give adiabatic evaporation. Adiabatic evaporation is not appropriate for large scale production because it makes it impractical to re-circulate the carbon dioxide. We have successfully tried two differing techniques for arranging the evaporation and for each technique the optimum operating conditions are slightly different.

Brief Summary Text (28):
In the second technique, the gaseous carbon dioxide from the evaporator is subsequently condensed and recycled through the column of hops. When the carbon dioxide is recycled in this way it is advantageous to use the heat obtained from the condenser in the evaporator e.g. by using a heat pump. In this kind of system it is preferred to operate with as nearly as possible a constant temperature throughout the circulating system in order to operate at a high thermal efficiency. It is, thus, preferable to operate at somewhat higher temperatures than are preferred in the first method described above despite the increase in pressure. In this technique the preferred extraction temperature is about 10.degree. C. (e.g. between 9.degree. and 12.degree. C.). At this temperature the extract is obtained in a sufficiently fluid form not to block the evaporator and the extraction temperature corresponds approximately to the peak solubility of .alpha.-acids in liquid CO.sub.2. This seems to favour slightly quicker extraction. Additionally performing the evaporation at a temperature of at least 10.degree. C. prevents any difficulty arising from the formation of solid carbon dioxide hydrate (CO.sub.2 -8H.sub.2 O) in the evaporator. At the pressures used carbon dioxide hydrate may form as a solid in the evaporator if the temperature is less than its melting point of about 10.degree. C.

Brief Summary Text (42):
In a particularly advantageous form the invention provides a method of making a stable hop extract of high quality which method comprises passing liquid carbon dioxide through a column of hops at a temperature of from -5.degree. C. to +15.degree. C. preferably at about 10.degree. C., evaporating the liquid carbon dioxide to obtain an extract of high purity in an inert vessel, condensing the evaporated carbon dioxide and recycling the liquid carbon dioxide through the hops to continue the extraction until at least a substantial proportion, preferably at least 70%, of the .alpha.-acids present in the hops are extracted but halting the extraction of the said column of hops before any substantial amount of undesired impurities are extracted from the hops.

Detailed Description Text (14):
2.0 Kg of powdered Wye Saxon hops containing 8.9% of .alpha.-acids were placed in the column (5 cm ID.times.180 cm) of a semi-continuous extraction apparatus generally as illustrated in FIG. 3. Liquid carbon dioxide was circulated through the system at a rate of 6.5 liters/hour for 3 hours. The temperature of the liquid carbon dioxide inside the column was about -5.degree. C. After 3 hours 282 g of extract had been obtained.

Detailed Description Text (15):
The extract was examined by thin layer chromatography using the procedure described by the European Brewery Convention (J. Inst Brewing, 1970, 76, 386) and the presence of only .alpha.-acids and .beta.-acids were revealed as two distinct spots when the plate was sprayed with methanolic ferric chloride reagent (1% w/v). When products, obtained by extracting hops with organic solvents are examined by this technique the chromatograms are normally complex and often consist of more than ten spots. The extract was shown to contain 56.7% of .alpha.-acid when estimated by a conductometric procedure (J. Inst Brewing, 1970, 76, 343) using methanolic lead acetate. Hence 89.9% of the available .alpha.-acids were extracted from the hops using liquid carbon dioxide. Examination of the extract by column chromatography on Sephadex (J. Inst Brewing, 1972, 78, 57) revealed that 83.0% of the .beta.-acids present in the hops had been extracted.

Detailed Description Text (22):
(b) 94.8% of the available .alpha.-acids and 76.2% of the available .beta.-acids were extracted by the liquid carbon dioxide.

CLAIMS:

1. A method of making a high purity hop extract comprising .alpha.-acids, .beta.-acids and hop oil, and having no more than 0.5% tannin and 0.5% hard resins, which consists essentially of passing liquid carbon dioxide at a temperature of from -5.degree. C. to +15.degree. C. through a column of hop material to extract at least a portion of the .alpha.-acids contained in the hops into the liquid carbon dioxide and subsequently evaporating the carbon dioxide, in equipment which is chemically inert to the extract, to thereby recover said hop extract of high purity.

4. The method as claimed in claim 1, wherein the evaporated carbon dioxide is condensed and re-circulated through the column of hops.

7. A method of making a golden yellow, high purity hop extract which consists essentially of passing liquid carbon dioxide at a temperature of from -5.degree. C. to +15.degree. C. in an upward direction through a substantially vertical column of hop material, the ratio of length to diameter of the column being from 4 to 1 to 50 to 1, to extract at least a portion of the .alpha.-acids contained in the hops into the liquid carbon dioxide and subsequently evaporating the carbon dioxide, in equipment which is chemically inert to the extract, to thereby recover said golden yellow, high purity hop extract having the following composition in weight percent:-

.alpha. acids: 40 to 75

.beta. acids: 20 to 40

Total resins (including .alpha. and .beta. acids): 70 to 98

Hop oil: up to 10

Water: up to 5

Uncharacterized soft:

resins: up to 5

Hard resins: up to 0.5

Tannins: up to 0.5

Chlorophyll: up to 0.2

Fine solids: up to 0.5

Inorganic salts: up to 0.5

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L7: Entry 34 of 44 File: USPT Aug 1, 1978

DOCUMENT-IDENTIFIER: US 4104409 A
TITLE: Production of hop extracts

Abstract Text (1):
Process for extraction of hops utilizing supercritical gases such as carbon dioxide.

Brief Summary Text (9):
A process has now been discovered for the production of hop extracts in which air dried hops are extracted with extraction solvents in the supercritical state with respect to temperature and pressure. Suitable solvents include carbon dioxide, saturated and unsaturated hydrocarbons containing up to about three carbon atoms; preferably those in which at least fifty percent of the hydrogen atoms of the parent hydrocarbon are replaced with halogen; sulfur hexafluoride; nitrous oxide and mixtures of these having a critical temperature of from about 30.degree. to 100.degree. C. The extracts are separated from the solvent gas by reducing the pressure or temperature or both to below the critical points.

Brief Summary Text (10):
Carbon dioxide is the most preferred extraction medium, but others such as SF.sub.6, CHF.sub.3, CF.sub.3 Cl, CF.sub.2 .dbd. CH.sub.2, CHF.sub.2 Cl, C.sub.3 F.sub.8, CH.sub.2 .dbd.CH.sub.2, CH.sub.3 CH.sub.3 and N.sub.2 O either singly, mixed with each other or with carbon dioxide can be employed. Supercritical CO.sub.2 in the sense of this invention is CO.sub.2 the temperature of which is above the critical temperature of CO.sub.2 (31.3.degree. C), while its pressure is above the critical pressure (about 73 atmospheres). Other supercritical gases used in the invention may be similarly described. Typically, the critical pressure will be at least 70 atmospheres and the temperature at least 30.degree. C.

Detailed Description Text (1):
For illustrative purposes, the invention will be principally described with reference to the most preferred solvent carbon dioxide. The presently preferred process will be best understood by reference to FIG. 1.

Detailed Description Text (2):
The gas used in the process is stored in tank 1 controlled by valve 2. In carrying out the method of the invention, the entire system is flushed with dry CO.sub.2 and valves 3, 4, 5 and 6 are opened. All other valves are closed. Liquid carbon dioxide flows from tank 1 to liquid gas pump 11 and then into heat exchanger 12 where it is brought to supercritical conditions of temperature and pressure. The resulting gas flows through valve 7 which is opened and into pressure pipe 13 which is formed with heat jacket and equipped with filter plates having a filter threshold of five millimicrons to support the hops.

Detailed Description Text (14):
In another modification of the method, the hops liberated of the total resin and the essential oils by the treatment with dry, supercritical CO.sub.2, cannot be treated directly with water, but first in a second step with wet, supercritical CO.sub.2. The wetting of the gas current is effected in tank 19, which is cut into the circuit through valves 8 and 9. There is thus obtained an extract fraction whose constituents have some tannin character. The water extract thus obtained, which is either dry or hydrous, as desired, is then added to the extract obtained in the

first step and can then be used either alone or mixed with the extract portion obtained from the strictly aqueous extraction (third step), which can be either spray- or freeze-dried.

CLAIMS:

4. A process as in claim 1 wherein the extraction gas is carbon dioxide.

5. A process according to claim 1 in which the extraction solvent gas is dry carbon dioxide and resin and essential oils are thereby extracted from the hops in a first extraction step, and the extracted hops are further extracted with wet supercritical carbon dioxide, in a second extraction step whereby tannin is extracted from the hops.

13. A process according to claim 1 wherein the extraction gas is selected from the group consisting of carbon dioxide; saturated and unsaturated hydrocarbons containing up to about three carbon atoms; nitrous oxide; saturated and unsaturated halogenated hydrocarbons containing up to about three carbon atoms; and mixtures of these.

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